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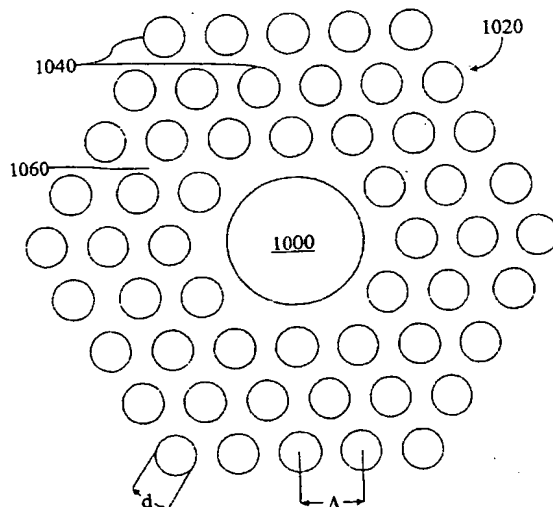
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(54) Title: FLUID ANALYSIS USING PHOTONIC CRYSTAL WAVEGUIDE



(57) Abstract: Fluid analysis may be achieved according to embodiments of the present invention by introducing a water-based analyte into a hollow core region (1000) of a photonic crystal waveguide, for example a photonic crystal fibre, acting as a liquid core waveguide. The waveguide has, around the core region, a microstructured cladding region (1020), which comprises a silica matrix (1060) filled with a periodic array of air holes (1040). The waveguide is able to guide a wide range of wavelengths of light, including excitation and sensing wavelengths of light, through the analyte by total internal reflection in the core region. This is possible because the cladding region of a photonic crystal waveguide can be arranged to have an effective refractive index lower than water, even though silica has a refractive index higher than water. Many other analytes, with even lower refractive indices than water, may be tested according to other embodiments of the present invention.

WO 2004/001465 A1

## FLUID ANALYSIS USING PHOTONIC CRYSTAL WAVEGUIDE

Technical Field

The present invention is in the field of optical sensing and relates in particular, but not  
5 exclusively, to optical sensing using liquid core waveguides.

Background Art

It is known to use optical waveguides, for example fibres, in spectroscopy experiments for studying the structure, composition and dynamics of fluids through  
10 absorption, emission and scattering of light. As used herein, the term "light" means electromagnetic radiation of any wavelength, for example, in the infra-red, ultra-violet or visible part of the electromagnetic spectrum.

One known way of using an optical waveguide in spectroscopy experiments is by introducing an analyte, typically a liquid, into a hollow core of the waveguide, sometimes  
15 known as a liquid core waveguide (LCW), and analysing light propagating through the analyte, or emanating from the analyte, for example, due to some chemical reaction involving the analyte. For example, a LCW may be filled with an analyte and exposed to light of an appropriate wavelength (or wavelength band) from a light source. Light coupled back out of the LCW may be tested in a number of ways depending on what substance, or measurand,  
20 is being measured. For example, absorption, changes of the optical index, non-linear effects such as Raman and/or Brillouin scattering and fluorescence (intensity, spectrum, or lifetime) may be measured using a LCW and appropriate light sources, detectors and, where necessary, reagents.

Clearly, particularly where long interaction lengths between fluid and light are  
25 desirable, it is important that a LCW is able to guide light along a desired interaction length sufficiently well to permit meaningful excitation, light recovery and analysis.

It is possible to use a LCW made from silica glass, which has desirable and well-understood optical properties. However, most fluids of interest have refractive indices lower than silica and, as such, waveguides filled with such fluids would not support light guidance  
30 in the fluid core by total internal reflection.

Additionally, there are few materials that possess a refractive index lower than that of water, which is around 1.33 in the visible part of the spectrum, where water is probably the most widely used analyte solvent.

Many alternatives to simple glass capillaries have been proposed, for example:  
35 internally-coated glass capillaries; externally-coated glass capillaries; and polymer capillaries

to name just a few. Each variant typically has advantages and disadvantages over silica glass.

One group of amorphous fluoropolymer materials has been reported as being suitable for use as LCW and are sold under the name "Teflon® AF". Teflon® AF is sold by  
5 DuPont™ and has significant optical clarity and relatively low refractive indices; down to around 1.29 – see, for example, <http://www.dupont.com/teflon/af/>. Due to its relatively low refractive index, Teflon® AF LCW can be used for testing many liquids, including water.

One example of using a Teflon® AF LCW is described in R.D. Waterbury, W. Yao, and R.H. Byrne, "Long Pathlength Absorbance Spectroscopy: Trace Analysis of Fe(II) Using  
10 a 4.5 m Liquid Core Waveguide", *Analytica Chimica Acta*, vol. 357, pp. 99-102, 1997. The paper describes a technique for measuring the ferrous iron (Fe(II)) content of a water sample using a 4.47m long Teflon® AF LCW filled with a water sample. The absorption peak of ferrozene complex (Fe(FZ)<sub>3</sub>), as used in the experiments, coincides with the 480-700nm transmission window for water, thus minimising the extent of light absorption by the water.

15 Another example of using a Teflon® AF LCW is described in Mark Holtz, Purnendu Dasgupta, and Genhfa Zhang, "Small-Volume Raman Spectroscopy with a Liquid Core Waveguide", *Analytical Chemistry*, vol. 71, no. 14, pp. 2934-2938, 1999. In this paper the Teflon® AF LCW is used as a Raman waveguide detector in liquid chromatography experiments on test mixtures such as benzene, carbon tetrachloride and aqueous solutions  
20 of 2-propanol. In all cases illumination of the LCW was transverse to its axis with end-on detection of Raman-scattered light through a transparent window at the LCW terminus. Transverse illumination minimises the amount of excitation light that reaches the light detector since light entering the core of the LCW outside of the total internal reflection acceptance angle simply emerges from the LCW walls and does not reach the detector.  
25 Additionally, transverse illumination reduces the level of detected Raman scattered light caused by the LCW walls, which is desirable. On the other hand, Raman scattering in the analyte occurs in every direction so at least a portion of the Raman-scattered light will reach the detector.

A further example of using a Teflon® AF LCW is described in Jianzhong Li, Purnendu  
30 K. Dasgupta, Zhang Genfa, "Transversely illuminated liquid core waveguide based fluorescence detection. Fluorometric flow injection determination of aqueous ammonium/ammonia." *Talanta* 50 (1999) 617-623. In this paper, an OPA-sulphite-NH<sub>3</sub> fluorometric detection system is used to measure the concentration of ammonia in water. The analyte is premixed, pumped into a Teflon® AF LCW and exposed to light from a  
35 miniature Hg backlight. Fluorescence resulting from exposure to the light is detected by a blue-sensitive photo-detector.

While Teflon® AF proves better than silica glass as a LCW material, in the respect that it is able to support total internal reflection at the analyte-inner wall boundary, it is significantly more expensive than silica glass and less temperature resistant and mechanically stable in mechanical applications.

5

#### Disclosure of the Invention

Photonic crystal fibres (PCF) (also sometimes known as 'holey fibres' or 'microstructured fibres') have in recent years been developed and typically comprise a cladding made of transparent matrix material with plural air holes extending longitudinally  
10 through the matrix material, along the length of the fibre and surrounding a core.

In one example, a PCF can be made entirely of fused silica glass with a solid core. By careful selection of core size, cladding hole size, spacing and geometry it is possible to make such a fibre index guiding in the core due to there being an 'effective refractive index' in the cladding that is lower than the refractive index of the core (see, for example, US Patent  
15 6,334,019, Birks et al.)

In another example, a PCF can guide light by virtue of a photonic bandgap effect, rather than by total internal reflection, when the effective refractive index of the cladding is higher than the refractive index of the core. In effect, light may be confined inside a hollow core due to a bandgap formed by suitably arranged air holes extending through a cladding  
20 matrix material and surrounding the core (see, for example, "Single-mode photonic band gap guidance of light in air", Science 285 p.1537 (1999) or WO00/60388).

The core of a bandgap guiding PCF may be under vacuum, filled with air or some other low index material. Bandgap guidance in PCFs is also, in principle, possible when the core refractive index is higher than the cladding effective refractive index, although index  
25 guidance will contribute to (and probably dominate) bandgap guidance under such conditions.

For index guidance, or guidance by total internal reflection, in a PCF the holes in the cladding may be regularly arranged and of equal size. Alternatively, the holes may vary in size and/or be arranged in a non-uniform, irregular or non-periodic way (see, for example,  
30 WO00/49436). In any case, the cladding holes may be air-filled or filled with other material(s) and, in the case of index guiding PCFs, the core may be of a different material to the cladding matrix material; as long as the effective refractive index of the cladding is lower than the core refractive index.

In arriving at the present invention, the inventors have appreciated that certain PCF  
35 structures can be used in certain optical sensing arrangements.

According to a first aspect the present invention provides a sensor comprising a waveguide having a hollow core region extending through a cladding region, the cladding region comprising a matrix material having a first refractive index and, formed in the matrix material, a plurality of elongate, longitudinal holes surrounding the core region, the waveguide providing, at a sensing and/or excitation wavelength of light, an effective refractive index in the cladding region that is lower than the refractive index of an analyte to be tested such that the waveguide is index guiding with the analyte in the core region.

While the term "analyte" typically imports the limitation of being a liquid, it is not inconceivable that the present invention may find application for certain gaseous or, indeed, other, non-liquid analytes and, as such, the term "analyte" as used herein should not be restricted to meaning liquids alone; as long as the waveguide supports index guidance of light in the presence of the analyte.

As used herein, the term "waveguide" is intended to include structures that are relatively flexible, for example optical fibres, and structures that are relatively rigid. In keeping with this, although the term PCF is typically associated with relatively flexible optical fibre waveguides, as used herein the terms 'photonic crystal waveguide' and 'PCF' may be used interchangeably, depending on context, to describe both or either flexible and rigid waveguide structures.

The ability to manufacture a sensor that includes a waveguide that guides light by total internal reflection in the core when a significant portion of the cladding material (or all of it where the holes are filled with air or under a vacuum) need not have a refractive index lower than the analyte, opens up potential for using a whole new class of materials, for example silica glass, that have had the required optical transmission properties, for example optical transparency, but have typically not had a refractive index lower than many practically relevant analytes to be tested.

The waveguide may provide, at sensing and excitation wavelengths of light, an effective refractive index in the cladding region that is lower than the refractive index of an analyte to be tested. The sensing and excitation wavelengths of light may be in the same or different regions of the electromagnetic spectrum. Advantageously, photonic crystal fibre waveguides may be made which can guide different wavelengths of light, even at significantly different wavelengths, in a single mode.

The sensor may comprise a waveguide that is bandgap guiding. Bandgap guidance may be in the presence or absence, or both presence and absence, of the analyte. As such, light may be strongly guided by the entire length of the waveguide even when the analyte only fills a part or parts of the core along its length.

The cladding holes may have a refractive index that is lower than the refractive index of the matrix material. For example, the cladding holes may be filled with air, or another material, or gas, or be under vacuum, depending on the effective refractive index that is required. The effective refractive index of the cladding of the waveguide will typically be below 1.45, the approximate index of fused silica glass, and will typically be well below 1.45, for example 1.29 or lower. As such, the waveguide can even be designed to be index guiding in the presence of light petro-chemicals, or even liquefied gases, in the core.

For most applications it is anticipated that air-filled holes would be appropriate. As used herein, the term "hole" typically means an elongate cylindrical volume running along the length of the waveguide and a hole may have a circular or non-circular (for example, elliptical, triangular, square or hexagonal) cross section. In some embodiments, it is anticipated that the geometry of a hole may vary along the length of the waveguide or even be disjoint in that it ends or begins part way along the waveguide. Additionally, or alternatively, a hole or holes may run parallel to or form a helix about a core region. Generally, unless otherwise stated, the term "hole" should not be taken to limit the volume's geometry, or limit the material that fills the volume, as long as the waveguide functions according to the present invention.

Sensors according to embodiments of the present invention may have a waveguide core diameter less than 50 $\mu$ m, 20 $\mu$ m, 10 $\mu$ m or even less than 5 $\mu$ m. Using known PCF manufacturing techniques, as described below, permits a wide range of core diameters to be made. This ability means that one has a significantly greater control over the propagation characteristics of a PCF waveguide than one would have over, for example, other known LCW and, accordingly, much smaller analyte volumes can be used if so desired.

In some embodiments, preferably, light guidance by the waveguide is limited to one, or only a few, modes at the sensing and/or excitation wavelength. More preferably, in some embodiments, the waveguide guides light at the sensing and/or excitation wavelength in only a single mode. In some embodiments, the waveguide guides light at the sensing and excitation wavelengths of light in only a single mode with the analyte in the core region. The ability to make waveguides having very small core diameters is helpful in this respect. Single mode operation means there is no modal dispersion facilitating, for example, spatially resolved measurements along the axis of the fibre by using known optical time domain reflectometry (OTDR) or optical coherence domain reflectometry (OCDR) techniques. Further, experiments can be highly repeatable in that they are unaffected by the presence of higher order modes.

In some embodiments, the sensor waveguide has cladding holes that are sealed at one or both ends. Accordingly, it is impossible for the analyte to accidentally enter the

cladding holes when analyte is introduced into an end of the waveguide at which the cladding holes are sealed. The holes may be sealed by appropriate heat treatment to collapse the ends of the holes, or by applying a material such as glue, silicone or even wax to plug the ends of the holes.

5 In some embodiments, at least part of the inner surface of the core region is coated with, or has deposited thereon, a reactant for causing detectable optical changes in the analyte. Then, when the reactant comes into contact with certain compounds that are (expected to be) present in an analyte fluorescence or absorption occurs. Use of such reactants is known, for example, in connection with certain biosensors. For example, the  
10 reader is referred to US 2002/0009719 in which reactants comprise nucleic acids which are deposited onto the distal ends of standard optical fibre strands arranged into a unitary array of fibre strands. The nucleic acids are used to detect different oligonucleotide fragments. As explained in the aforementioned patent, there are numerous techniques for accurately depositing reactants and such techniques may easily be applied to depositing reactants on  
15 and in PCF cores. Certain techniques for depositing reactants may involve vapour phase deposition or evaporation.

In some embodiments, the sensor comprises a light detector arranged to receive light coupled from the waveguide.

For example, in some embodiments, light may be coupled from one end (e.g. the  
20 proximal end) of the waveguide. The detector could, for example, be a single diode, a diode array, or a spectrometer depending on the nature and wavelength(s) of light that is/are to be measured.

In alternative embodiments, light emanating from the core region may be coupled from the side of the waveguide. An exemplary detector may comprise a charge coupled  
25 device (CCD) array, for detecting light emanating from the side of the waveguide along a significant length (for example, several millimetres or even centimetres) thereof. Such an embodiment may, for example, comprise a source, light from which is coupled into an end of a waveguide filled with analyte, and the detector may be arranged to detect fluorescence from the analyte along a portion of the waveguide.

30 In some embodiments, the sensor further comprises an excitation source for exciting the analyte. The excitation source may comprise a light source arranged to couple light into the waveguide. The light may be required in order to stimulate, for example, Raman scattering or fluorescence in the analyte, both of which would typically be detected by a light detector at the proximal end of the PCF. The analyte may be excited by introducing light into  
35 the waveguide from a direction transverse to the longitudinal axis of the waveguide. Alternatively, the light source may be arranged to couple light into a distal end or the

proximal end of the waveguide. Means other than light, for example RF radiation, a chemical reaction, an electric field or radioactive particles, may be used to stimulate the analyte.

In some embodiments, the light may be required to pass through the analyte, such that certain wavelengths of the light may be absorbed by compounds that are expected to be present in the analyte, in order that the absorption spectrum of the analyte can be determined. As such, it may be preferable to couple light into the distal end of the waveguide, or into the proximal end of the waveguide. Of course, such end coupling of light could be used in, for example, fluorescence and scattering experiments as well, since the direction scattered or fluorescent light propagates in a waveguide does not necessarily depend on the direction of propagation of excitation light.

The sensor may comprise a coupler, arranged to couple light into and/or from the same end of the waveguide. For example, the coupler may be arranged to couple light from a light source into a proximal end of the waveguide and also couple light from the proximal end of the waveguide into a light detector. The coupler may conveniently be a fused fibre coupler having first port coupled to the light source, a second port coupled to the light detector and a third port coupled to the proximal end of the waveguide.

The sensor may comprise a reflector that is arranged to reflect light that is propagating towards the distal end of the waveguide back towards the proximal end of the waveguide. The reflector may be arranged to selectively reflect light other than light from the light source.

In some embodiments, the waveguide comprises plural core regions, at least one core region being a hollow core region. Preferably, the sensor comprises plural hollow core regions and, more preferably, at least part of the inner surfaces of plural hollow core regions are coated with a reactant for causing a detectable optical change in the analyte. Different core regions may be coated with different reactants for causing different detectable optical changes in the analyte.

Such an arrangement might find application as an alternative to the kind of arrangement described in US 2002/0009719 (as described above), which describes use of an array of bundled, solid core optical fibres, the distal end of each fibre being bound to a different nucleic acid, to sequence DNA. According to embodiments of the present invention, one or more photonic crystal waveguides having multiple hollow cores each coated internally with a different nucleic acid could be used in place of an array of separate optical fibre strands.

Preferably, the sensor has a photonic crystal waveguide comprising fused silica glass and, advantageously, only fused silica glass. As such, the waveguide may be manufactured with relatively insignificant material cost compared with, for example, Teflon® AF, while



exhibiting chemical inertness, high temperature stability for cleaning, contamination free fabrication, and easy handling (for example for cleaving). Of course doped silica or, other materials, for example other kinds of glasses and polymers, may be used instead or in combination with silica.

5 Another advantage of providing a sensor with a waveguide that comprises only fused silica glass is the dramatically increased lifetime of the waveguide when used with short wavelength light, for example UV-light, which is commonly used in fluorescence sensing. Other materials, particularly doped glass materials and some polymers, undergo significant and rapid degradation when exposed to UV-light rendering the waveguide useless after only  
10 a relatively short period of time.

According to a second aspect, the present invention provides a photonic crystal waveguide for use in an analyte sensing arrangement with an analyte having an analyte refractive index, the waveguide comprising a core region and a cladding region, the cladding region comprising a matrix material having a first refractive index and embedded along its  
15 length, parallel to a core region, an array of cladding holes surrounding the core region, the waveguide providing, at a sensing and/or excitation wavelength of light, an effective refractive index in the cladding region that is lower than the refractive index of the analyte such that the waveguide is index guiding with the analyte in the core region.

The core region of the photonic crystal waveguide may be at least partially filled with  
20 analyte.

According to a third aspect, the present invention provides a method of sensing a property of an analyte including the steps:

introducing the analyte into at least a part of the core region of the photonic crystal waveguide described above;

25 coupling light into or otherwise exciting the analyte; and  
detecting light coupled from the waveguide.

Other aspects and embodiments of the invention will become apparent from the following description and claims.

### 30 Brief Description of the Drawings

Embodiments of the present invention will now be described by way of example only with reference to the accompanying drawings, of which:

Figure 1 is a schematic representation of the transverse cross section of an exemplary hollow core PCF structure.

35 Figure 2 is a graph showing how effective refractive index in the cladding of a PCF varies with the air filling fraction of the cladding and the optical wavelength;

Figure 3 is a schematic diagram of apparatus for carrying out Raman scattering or fluorescence experiments;

Figure 4 is a schematic diagram of apparatus for carrying out absorption experiments;

Figure 5 is a schematic diagram of apparatus for carrying out fluorescence experiments;

5 Figure 6 is a schematic diagram of apparatus for carrying out sensing wherein both a light source and a light detector are coupled to the same end of a photonic crystal waveguide;

Figure 7 is a schematic diagram illustrating index guidance (Region A) and photonic bandgap guidance (Region B) in a single photonic crystal waveguide;

10 Figure 8 is a schematic diagram of a multi-core photonic crystal fibre which finds application in embodiments of the present invention;

Figure 9 is a diagram of a practical photonic crystal waveguide that finds application in sensors according to one or more embodiments of the present invention; and

15 Figure 10 is a mode field plot at  $0.75\mu\text{m}$  for a water-based analyte introduced into the core of the photonic crystal waveguide of Figure 9.

#### Best Mode For Carrying Out the Invention, & Industrial Applicability

Figure 1 is a schematic representation of the transverse cross section of an exemplary hollow core PCF structure. The hollow core region 1000 is surrounded by a  
20 cladding region 1020 comprising a substantially periodic structure of air holes 1040 embedded in a matrix material 1060 and surrounding the core region 1000. It will be appreciated that only the first few layers of air holes are shown, for clarity reasons only, and that typically in practice the core 1000 will not be perfectly circular, having deformations coinciding with the inner cladding holes around the periphery of the core region. The holes  
25 1040 in the cladding region are arranged in six-fold symmetric fashion about the core region 1000 and the core region is equivalent in size to seven cladding-sized air holes. This kind of PCF structure is described in detail in WO 00/60388 as being capable of guiding light in the hollow core region by virtue of a photonic bandgap in the cladding material. Embodiments of the present invention, which require that light be guided in a LCW by both bandgap and  
30 index guiding mechanisms, may benefit from use of this kind of PCF, as will be described in more detail below. However, it is emphasised that guiding by bandgap effects is not an essential element of the present invention.

For the PCF to behave as a LCW according to embodiments of the present invention, light needs to be guided along at least part of the waveguide by total internal reflection (for  
35 example, see region A in Figure 7). Clearly, the structure illustrated in Figure 1 will not guide by total internal reflection since the refractive index of the hollow core region 1000 is lower

than the effective refractive index of the cladding region 1020, where effective refractive index will be between those of air and the cladding matrix material 1060. However, by introducing a fluid into the core region 1000, it is possible to achieve light guidance by total internal reflection as long as the effective refractive index of the cladding region 1020 is lower than the refractive index of the fluid.

In Figure 1, the dimension  $d$  represents the diameter of a cladding hole and the dimension  $\Lambda$  represents the spacing, or pitch, between cladding hole centres.

Knowing the refractive index of air,  $n_{\text{air}}$ , and the refractive index of the cladding matrix material,  $n_{\text{cl}}$ , the skilled person is able to design a PCF having a cladding effective refractive index,  $n_{\text{eff}}$ , which is lower than the refractive index,  $n_{\text{fl}}$ , of a fluid to be tested. For example, the skilled person would be aware of the paper entitled "Endlessly single-mode photonic crystal fibre", Optics Letters, Volume 22, No. 13, pp961-963, July 1, 1997, which describes how to calculate effective refractive index on the basis of the an assumption that the cladding is an infinite uniform medium. According to that paper, a PCF guiding light by total internal reflection is equivalent to a standard optical fibre and has propagation constants  $\beta$  available to light in the core but not to light propagating in the cladding, such that:

$$kn_{\text{cl}} > \beta > \beta_{\text{FSM}} \quad (\text{Equation 1})$$

where  $k=2\pi/\lambda$ ,  $n_{\text{cl}}$  is the index of the cladding matrix material, for example fused silica glass, and  $\beta_{\text{FSM}}$  is the propagation constant of the fundamental space-filling mode (FSM). The FSM is the fundamental mode of the infinite photonic crystal cladding if the core region is absent, so  $\beta_{\text{FSM}}$  is the maximum  $\beta$  allowed in the cladding. Inasmuch as the lower limit of  $\beta$  in a standard step index fibre is  $kn_{\text{cladding}}$ ,  $n_{\text{eff}}$  can be shown to be:

$$n_{\text{eff}} = \beta_{\text{FSM}}/k \quad (\text{Equation 2})$$

The graph in Figure 2 shows the variation in effective refractive index,  $n_{\text{eff}}$  (on the y-axis) with normalised frequency,  $\Lambda/\lambda$  (on the x-axis) for 5 values of  $d/\Lambda$ , which are (from top to bottom)  $d/\Lambda = 0.2, 0.4, 0.6, 0.8, 0.9$ . The plots assume that the refractive index of silica is 1.44 and does not change with wavelength. The plots were calculated under the approximations described in the above-referenced Optics Letter.

The graph in Figure 2 indicates that as the air filling fraction (determined by  $d/\Lambda$ ) increases, within certain bounds, the effective refractive index of the cladding decreases for a given wavelength of light.

For example, where  $\Lambda$  is the same as  $\lambda$ ,  $d/\Lambda$  needs to be greater than around 0.6 for guiding by total internal reflection, in the case where the fluid is water having a refractive index of around 1.33.

Considering a specific example of fluorescence sensing using a water-based analyte  
5 irradiated with light in the UV range, at a wavelength around 400nm or less, the upper bound for pitch would be around 3.5 times the wavelength with a respective  $d/\Lambda$  of around 0.9. The graph shows that by decreasing the pitch it would be possible to decrease either the necessary  $d/\Lambda$  or the effective refractive index.

It will be appreciated that in some embodiments it is desirable for the light to be  
10 guided in only a single mode. For a standard fibre with core radius  $\rho$  to be single mode it is necessary for the fibre to have a  $V$  value less than 2.405, where  $V$  is:

$$V = (2\pi\rho / \lambda)(n_{\text{core}}^2 - n_{\text{cladding}}^2)^{1/2} \quad (\text{Equation 3})$$

15 By reference again to the paper entitled "Endlessly single-mode photonic crystal fibre", it will be seen that it is possible to calculate an equivalent  $V$  value,  $V_{\text{eff}}$ , for PCF with fluid in the core, as:

$$V_{\text{eff}} = (2\pi\rho / \lambda)(n_{\text{fl}}^2 - n_{\text{eff}}^2)^{1/2} \quad (\text{Equation 4})$$

20

where  $n_{\text{fl}}$  is the refractive index of the fluid and  $n_{\text{eff}}$  is the effective refractive index of the cladding, as defined in the above-referenced Optics Letter. Although 2.405 is not typically the cutoff for single mode operation in a PCF, single mode operation can be ensured by suitable choice of fibre parameters and wavelength. For example, for regular  
25 PCF where the core is silica (rather than a fluid), the cutoff  $V_{\text{eff}}$  is 4.1 (see, for example, T.A. Birks, D.Mogilevtsev, J.C. Knight, P.St.J. Russell, J. Broeng, P.J. Roberts, J.A. West, D.C. Allen and J.C. Fajardo, "The analogy between photonic crystal fibers and step index fibers" Proceedings of the Optical Fiber Communication Conference (OFC, San Diego, California), February 1999, pp.114-116).

30 On the basis of the above, the skilled person would be able to design a PCF which guides a required number of modes by total internal reflection through a particular liquid to be tested.

Embodiments of the present invention effectively use a hollow core PCF as a LCW, where a liquid to be tested is introduced into the hollow core and the cladding effective  
35 refractive index of the PCF is lower than the refractive index of the liquid. In cases where the cladding matrix material has a refractive index higher than that of the liquid, it is necessary

for the holes to have a refractive index that is lower than that of the liquid. In the example provided in relation to Figure 1, the holes are air-filled, and there is, therefore, a need to introduce the fluid into the core of a PCF while not introducing the same fluid into the cladding holes.

5        There are a number of ways available to the skilled person by which fluid can be introduced into the core but not the cladding holes. Obviously, the fluid can be introduced directly into only the core using a narrow tube between a fluid reservoir and the opening of the core. For small bore cores, however, direct filling may be fiddly or time-consuming and it may be better to use an alternative approach. For example, the PCF may be manufactured  
10 so that the ends of the holes in the cladding are sealed. Then, a connector could be attached over the entire end of the PCF with fluid entering only the core. Alternatively, the PCF may be heat treated after manufacture using, for example, a taper rig and appropriate heat source, whereby the cladding holes but not the core collapses at one or both ends. Such techniques are described in WO 00/49435 and need not therefore be described herein  
15 in any significant detail. For example, it will be appreciated that, if a region of a PCF is heated, surface tension dictates that smaller cladding holes tend to collapse faster than a larger core hole. As such, a region of a PCF can be heat treated so that all holes collapse and then the PCF can be cleaved sufficiently far from the entirely collapsed region that the core hole is revealed but the cladding holes are still closed.

20        Alternatively, the ends of the cladding holes may be 'plugged', for example, using silicone, wax or glue. Further still, it may be possible to 'strip' the cladding region away from the core region for a short distance away from the end of a PCF. Then, it is possible to dip that end of the PCF into a fluid reservoir in order to collect the fluid into the core while ensuring that the cladding holes, which have in effect receded from the end of the PCF, are  
25 not exposed to the fluid.

It is assumed for the purposes of the following embodiments that appropriate steps are taken to ensure that the liquid is introduced only into the core of the photonic crystal LCW and not into the cladding holes.

Figure 3 illustrates schematically exemplary apparatus for performing fluorescence  
30 experiments on a fluid. The apparatus would equally find application in Raman scattering experiments. The apparatus is based on prior art apparatus described in the Jianzhong Li et al. paper cited above and, as such, full details of the apparatus are not included herein. According to Figure 3, the apparatus comprises a hollow core photonic crystal waveguide acting as a LCW 3000. The cladding holes of the LCW 3000 are not illustrated in the  
35 drawing for reasons of clarity only, and it should be remembered that the cladding would in practice be microstructured. Below the bottom end of the LCW 3000 as shown is provided

an optical fibre 3020 which is optically aligned with, and has a similar core size as, the LCW 3000. A photo-detector 3040 receives light coupled from the LCW 3000, via the optical fibre 3020, and is connected to appropriate spectroscopy equipment (not shown) in order that received light can be measured or analysed in an appropriate manner.

5       The optical fibre 3020 is held in optical alignment with the LCW by an opaque tee fitting 3060, an arm 3080 of which also provides an orifice into which an inlet tube 3100 is inserted. A discharge tube 3120, through which excess analyte can escape to be discarded or recycled, is connected by a union 3140 to the top of the LCW 3000 as shown.

10       A majority of the LCW 3000 is housed within a stainless steel tube 3160, which has a highly polished inner surface. Also located within the tube 3160 is a lamp 3180 that is used to illuminate the analyte in the LCW 3000. Power leads 3200 for the lamp 3180 extend through a hole in the tube and are connected to an appropriate power supply 3220. The stainless steel tube 3160 has plural functions: it maximises exposure of the LCW 3000 to the light from the lamp 3180, it prevents light from other sources entering the LCW and adds  
15       strength to the overall LCW arrangement.

In use, the analyte to be tested is pumped into the inlet tube 3100, through the tee 3060 and into the LCW core using a peristaltic pump arrangement (not shown). Excess analyte is discharged from the discharge tube 3120 to be discarded or recycled. The analyte may or may not be continually pumped through the LCW during an experiment.

20       It will be appreciated that the analyte comprises a fluid to be tested which, if required, has been pre-mixed with necessary reagents in order that fluorescence occurs when the analyte is illuminated.

When the lamp 3180 is on and the analyte in the LCW is illuminated, fluorescence is generated as a result. The optical fibre 3020 receives the fraction of the fluorescence that  
25       propagates by refractive index guidance down the LCW 3000 as shown and couples the light into the light detector 3040.

Figure 4 illustrates schematically exemplary apparatus for performing absorbance spectroscopy.

According to Figure 4, the apparatus comprises a hollow core photonic crystal  
30       waveguide acting as a LCW 4000. As in Figure 3, the cladding holes of the LCW 4000 are not shown. At the bottom end of the LCW 4000 as shown is provided a first optical fibre 4020 which has a core size matched with the core of the LCW 4000 and is optically aligned at one end with the core of the LCW and at the other with a spectrometer 4040. The optical fibre 4020 is held in optical alignment with the LCW by a first opaque tee fitting 4060, an arm  
35       4080 of which also provides an orifice into which a discharge tube 4100 is inserted.

A second optical fibre 4120 is aligned with the core of the LCW 4000 and is held in place by a second opaque tee 4140, similar to the first tee 4080, which also has an arm 4160 that provides an orifice into which an inlet tube 4180 is inserted. The second optical fibre 4120 is coupled to a light source 4200.

5 In this embodiment, a stainless steel tube 4220 is optional, since there is no need to concentrate light into the wall of the LCW 4000. However, it is still desirable to use the tube 4220 to limit ambient light entering the LCW 4000 and to add additional strength to the LCW arrangement.

10 In use, broadband light is coupled into the top of the LCW 4000 from the light source 4200 via the second optical fibre 4120. The first optical fibre 4020 receives light that has propagated down through the analyte in the LCW 4000 by virtue of refractive index guidance and couples the light into the spectrometer 4040 for spectral analysis.

The embodiments illustrated with reference to Figures 3 and 4 typically use rigid photonic crystal waveguides having relatively short lengths, for example a few tens of 15 millimetres or centimetres. Of course, a waveguide can in practice be any required length depending on how long the light-fluid interaction length needs to be, since photonic crystal waveguides, be they rigid or flexible, are able to transmit light with extremely low loss. Indeed, in practice, loss will likely be dominated by loss due to the analyte rather than the intrinsic loss caused by the LCW.

20 Figure 5 illustrates a further exemplary embodiment of the present invention, which is relatively cheap and may find particularly beneficial application in situations where the LCW is in fact disposable. Due to the relatively cheap nature of fused silica glass PCFs, for example compared with Teflon-AF, disposable LCW may be a realistic option.

According to Figure 5, a LCW 5000 comprises a photonic crystal waveguide that is 25 held in a bush 5020. The bush 5020 is itself held in the neck of a 'bulb' 5040 formed from an opaque, resilient material such as rubber. A light detector 5060 is supported by a clamp 5080 that is attached to the bush 5020 such that the detector is held in optical alignment with the end of the LCW 5000 within the bulb 5040. The detector 5060 is connected to appropriate detection circuitry 5100 through a hole in the wall of the bulb 5040. The 30 arrangement is such that the inside of the bulb 5040 is substantially sealed from the outside other than through the core of the LCW 5000.

As shown in Figure 5, the LCW arrangement may be adapted for fluorescence experiments by being introduced into a stainless steel chamber 5120 that has a highly polished inner surface and a lid 5140 that has generally at its centre a hole sufficiently large 35 to permit the LCW 5000 but not the bush 5010 to pass through, such that the LCW remains suspended within the chamber 5120. The chamber 5120 also houses a light source 5140 for

exciting the LCW 5000, the light source being attached to an appropriate power supply 5160 through a further small hole in the lid 5140.

In use, liquid may be introduced into the bulb 5040 by squeezing it to expel some air, dipping the free end of the LCW 5000 into a reservoir containing a liquid to be tested (not shown) and releasing the bulb. In other words, the arrangement operates like a pipette to draw fluid from the reservoir and into the core of the LCW 5000. Fluid testing proceeds in the normal way by simply switching on the light source 5140 and measuring fluorescence detected at the light detector 5060.

Figure 6 illustrates an alternative sensor arrangement similar to the arrangement Figure 5. In Figure 6, a photonic crystal LCW 6000 is held in a bush 6010 with the core at the top end of a LCW 6000 being held in optical alignment with an optical fibre 6020, which is itself held in place by a ferrule 6040. The optical fibre 6020 passes through a bulb 6060 and meets a first port 6080 of a known kind of fused fibre coupler 6100 having a waist region 6120 where light may couple from one fibre to another and visa versa. A second port 6140 of the coupler 6100 is connected to a light source 6160 and a third port 6180 of the coupler is connected to a light detector 6200. A fourth port 6220 is unused and is terminated near the waist region 6120.

In use, an analyte is drawn into the core of the LCW 6000 as before and light from the light source 6120 travels to the second port 6140 of the coupler and is split so that some of the light passes out of the first port 6080 to the LCW 6000 and some light is lost to the fourth port 6220. The light that reaches the LCW 6000 can, for example, cause Raman scattering or fluorescence in the analyte, a fraction of the scattered or fluorescent light travelling back towards the coupler 6100 and is split between the second 6140 and third 6180 ports. Fluorescence or back-scattered light that passes into the third port 6180 is detected by the detector 6200 while the remainder returns to the light source 6120 and is rejected.

Figure 6 may be modified by aligning a mirror 6240 with the free end of the LCW 6000. The mirror 6240 may be positioned at the base of a chamber, for example. As such, light from the source that travels through the LCW 6000 is reflected back into the LCW and part of that light is received at the light detector 6200, or spectrometer. An advantage of this arrangement, for example for absorption experiments, is that light travels through the analyte sample twice, increasing the probability of absorption. In effect, the length of the LCW may be reduced while maintaining the same effective interaction length of the analyte.

The skilled person will appreciate that the embodiments illustrated in Figures 5 and 6 may be adapted in a number of ways. For example, where long interaction path lengths are required between the analyte and the light, the LCW could be in the form of a coiled PCF fibre designed such that bend loss is not a significant loss mechanism. Further, the bulb



structure may be replaced by a rigid chamber structure which incorporates, for example, a syringe mechanism that is used to draw fluid into the LCW; or a peristaltic pump may be used to draw the fluid into the core.

Embodiments of the present invention so far described may take advantage of the  
5 ability to guide light along a photonic crystal waveguide by total internal reflection, requiring an analyte to be introduced along the entire length of the core of the LCW. However, as has already been alluded to above, it is possible to design a PCF, which has a hollow core that guides light by virtue of a bandgap in the cladding, while, at the same time, it has a structure that guides light by total internal reflection when a liquid is introduced into the core. Such a  
10 waveguide finds particular application in embodiments of the present invention where it is undesirable, inconvenient or unnecessary to introduce the liquid along the entire length of the LCW.

For example, referring to the arrangements shown in Figures 5 and 6, it would be relatively difficult to know when the analyte had been drawn along the entire length of the  
15 LCW, since the top end as shown of the LCW is obscured by the bush and bulb arrangement. As such it would easily be possible to introduce too much or too little liquid into the LCW: too much leading to spillage into the bulb and possible contamination of the detector, and too little leading to potentially significant loss of light guidance beyond the liquid-air interface. One way around this problem would be to provide a LCW that guides  
20 light of an appropriate wavelength in a hollow core by virtue of a bandgap effect in the cladding while also guiding light at the same wavelength by total internal reflection when the fluid is in the core. Then, liquid need only be introduced part way into the core while light still propagates along the entire length of the core. Indeed, in embodiments of the present invention where interaction length between an analyte and light within a LCW does not need  
25 to be long, it would be possible to rely on capillary action for introducing liquid only a relatively short distance into the core of a LCW. The effectiveness of this technique would clearly depend on the surface tension of the liquid, the bore diameter of the hollow core and the required interaction length.

The diagram in Figure 7 is a highly idealised two-dimensional representation of a  
30 photonic crystal waveguide 7000 having a hollow core region 7020 and a microstructured cladding region 7040. For clarity reasons only, the waveguide 7000 is shown as having only two layers of air holes 7060 in the cladding region 7040 either side of the core region 7020. As illustrated, the core region is part-filled with an analyte 7080, in region A, and part filled with air 7100, in region B. Light guidance by total internal reflection within the core 7020  
35 throughout region A is denoted by the two ray lines 7120. Light guidance is shown to be due

to a photonic bandgap in region B by Bragg reflections 7140 in the cladding and air hole layers of the waveguide 7000.

A photonic crystal waveguide of the kind illustrated in Figure 7 is optimised for minimum loss at the analyte-air interface by designing the waveguide such that the fundamental mode propagating in region A is as near as possible the same as the fundamental mode propagating in region B. It will be appreciated that refraction of light at the analyte-air interface may cause a certain amount of loss. However, a concave meniscus formed at the analyte-air interface due to surface tension in the analyte assists in minimising loss by reducing the angle of incidence of the light as it meets the interface.

With reference again to Figures 5 and 6, instead of using a bandgap effect to support sensing while only partially filling the waveguide with analyte, it might be permissible to draw too much liquid into the LCW and simply allow excess fluid to collect below the rim of the bush within the bulb. Additionally, the light detector or fibre end could be protected from fluid contamination by a transparent screen positioned between the end of the waveguide and the detector. In fact, it might be permissible to draw sufficient liquid into the bulb that the detector is immersed in the liquid. In this latter case, light coupling efficiency between the detector and LCW core may even be improved due to there being no liquid-air interface.

Figure 8 is a diagram that illustrates a different kind of PCF structure which finds application in further embodiments of the present invention.

The PCF in Figure 8 comprises three hollow core regions 8000, into which an analyte may be introduced, each surrounded by cladding holes 8040 extending through a matrix material 8060. In practice, the structure would typically have far more cladding layers, which are not shown for clarity reasons only.

In use, the multiple core PCF illustrated in Figure 8 may be used, for example, in the context of any of the preceding embodiments. For example, light from a single source may be coupled into all hollow cores of the PCF shown in Figure 8. A portion, for example at the distal end, of the inside of each hollow core may be coated with, or have deposited thereon, a different marker which reacts with different compounds in an analyte. Each marker may fluoresce with different intensity, or not fluoresce at all, depending in which compounds are in the analyte. In this manner, multiple simultaneous tests may be carried out on the analyte, with the fluorescent light recovered from the waveguide being analysed in a spectrometer.

Alternatively, or additionally, excitation light from different sources may be coupled into different individual cores, giving rise to the possibility of enacting different simultaneous experiments.

In practice, the number and arrangement of hollow cores may be varied depending on need and the ability for the cores to be index guiding in the presence of the analyte. For

example, the arrangement of six hollow cores marked as 8000' are an alternative to the arrangement of three cores marked 8000.

A multiple-core PCF is described in detail in US 6,301,420. In this patent, each core is formed from a solid material and is surrounded by cladding holes that provide an effective  
5 refractive index that is lower than core refractive index, thus rendering each core index guiding. By applying the principles described herein, the PCF in the patent may simply be adapted by replacing one or more of the solid cores with a hollow core into which an analyte may be introduced.

Figure 9 is an image of an inner region of an exemplary single hollow core photonic  
10 crystal waveguide suitable for use in accordance with embodiments of the present invention. The grey inner region is a hollow core, filled with a water-based analyte. The black regions around the core are air holes and the white regions around the air holes are fused silica glass, which forms the fabric of a microstructured cladding. The cladding comprises a triangular lattice of generally hexagonal air holes and the core region is the equivalent of  
15 seven missing air holes; an inner air hole and the six surrounding air holes.

In Figure 9, the cladding air holes have a diameter  $d$  (between parallel flat sides) of approximately  $1.8\mu\text{m}$  and a pitch  $\Lambda$  of  $2\mu\text{m}$ ; giving a value for  $d/\Lambda = 0.9$ . This equates to an approximate fraction by volume of air in the cladding of 0.8. With reference to the graph in  
20 Figure 2, it can be seen, at point A, that the effective refractive index  $n_{\text{eff}}$  of the cladding is slightly less than 1.3 at a wavelength of around  $0.75\mu\text{m}$ ; where water is substantially optically transparent in the region of  $0.75\mu\text{m}$ . The diameter of the core is approximately  $5.4\mu\text{m}$ .

With a water-based analyte, therefore, having a refractive index of around 1.33, it is apparent that the waveguide will guide light by total internal reflection at around  $0.75\mu\text{m}$ . This is illustrated by the mode field plot shown in Figure 10, which shows an intense inner  
25 region of light filling the core region and spreading only slightly into the cladding region. The less intense, outer regions in the mode field plot, which coincide with the cladding of the waveguide, are found to be more than 100dB lower in intensity than core region; the intensity having been plotted in dB.

The mode field plot in Figure 10 was simulated, using the waveguide structure in, and  
30 parameters associated with, Figure 9, by solving Maxwell's vector wave equation for the structure, using known techniques. In brief, Maxwell's equations are recast in wave equation form and solved in a plane wave basis set using a variational scheme. An outline of the method may be found in Chapter 2 of the book "Photonic Crystals – Molding the Flow of Light", J.D. Joannopoulos *et al.*, ©1995 Princeton University Press.

35 Waveguide structures similar to the one in Figure 9, having a  $2\mu\text{m}$  pitch  $\Lambda$  and values of  $d/\Lambda$  between 0.88 and 0.96, were simulated and were all found to guide light at  $0.75\mu\text{m}$

through the same, water-based analyte, which indicates that the performance of the waveguide would not be significantly affected by manufacturing imperfections, which typically cause slight fluctuations in cladding hole diameter  $d$  and pitch  $\Lambda$ .

The waveguide structure shown in Figure 9 is also a photonic bandgap wave guiding  
5 structure, which can guide light at  $0.75\mu\text{m}$  in the hollow core. As such, as explained above, it is expected that light at  $0.75\mu\text{m}$  would be guided both through a water-based analyte, due to total internal reflection, and through any region of the waveguide where no analyte is present, by photonic bandgap effects.

The present inventors have made photonic crystal waveguides with values of  $d/\Lambda$   
10 greater than 0.96. This provides clear scope for either testing analytes having a refractive index well below that of water or increasing significantly above  $2\mu\text{m}$  the pitch of a waveguide to be used for testing water-based analytes. The ability to increase pitch in this manner might be useful, by increasing the ease with which an analyte may be introduced into a larger core. This is because core size increases proportionally (for the same number of missing air  
15 holes) with pitch and it is easier to introduce water into a larger core.

Indeed, in some embodiments, it may prove beneficial to increase the size of the core more significantly. For example, a so-called nineteen cell core waveguide structure may be made, which has a core equivalent to nineteen missing air holes; an inner air hole, the six air holes surrounding the inner air hole and the twelve air holes surrounding the six air holes. It  
20 is anticipated that even larger cores may be made using known manufacturing methods.

Photonic crystal waveguides may be manufactured by known stack and draw techniques, whereby, first, a number of silica glass capillaries and rods are arranged into a pre-form that, on a macro scale, resembles the required waveguide transverse cross section. Then, the pre-form is heated in a fibre drawing tower and drawn into a waveguide having the  
25 required configuration and dimensions. See, for example, the paper by Birks et al., "Photonic Crystal Fibres: An endless Variety", IEICE Transactions on Electronics, Volume E84-C, No.5, May 2001 for further detail on manufacturing techniques.

A significant advantage of using photonic crystal waveguides in place of known LCW is that, since a photonic crystal fibre can be designed to be single-mode over a very wide  
30 bandwidth, sensing measurements will be highly repeatable, in the sense that they remain unaffected by the presence of higher order guided modes.

A further general advantage of using photonic crystal waveguides is that, due to the way they are typically manufactured (as described above), the inner surfaces of the cladding holes and, for a LCW, the core are extremely clean and free from any contaminants. As  
35 such, experiments benefit from the absence of the need to clean the inner surfaces of the photonic crystal waveguide.

The skilled person will appreciate that the arrangements described above, particularly as illustrated in Figures 5 and 6, can provide a highly compact, potentially disposable, (bio-) chemical sensing probes with potential to exhibit excellent optical properties and good overlap between the light and the analyte.

## CLAIMS

1. A sensor comprising a waveguide having a hollow core region extending through a cladding region, the cladding region comprising a matrix material having a first refractive index and, formed in the matrix material, a plurality of elongate, longitudinal holes surrounding the core region, the waveguide providing, at a sensing and/or excitation wavelength of light, an effective refractive index in the cladding region that is lower than the refractive index of an analyte to be tested such that the waveguide is index guiding with the analyte in the core region.
2. A sensor according to claim 1, wherein the waveguide provides, at sensing and excitation wavelengths of light, an effective refractive index in the cladding region that is lower than the refractive index of an analyte to be tested.
3. A sensor according to claim 1 or claim 2, wherein the waveguide is bandgap guiding.
4. A sensor according to any one of claims 1 to 3, wherein the cladding holes have a refractive index that is lower than the refractive index of the matrix material.
5. A sensor according to claim 4, wherein the cladding holes are filled with air.
6. A sensor according to any one of the preceding claims, wherein the core region diameter is less than 50 $\mu$ m.
7. A sensor according to any one of the preceding claims, wherein the waveguide guides light at the sensing and/or excitation wavelength of light in only a single mode with the analyte in the core region.
8. A sensor according to any one of the preceding claims, wherein the waveguide guides light at the sensing and excitation wavelengths of light in only a single mode with the analyte in the core region.
9. A sensor according to any one of the preceding claims, wherein the cladding holes are sealed at one or both ends of the waveguide.

10. A sensor according to any one of the preceding claims, wherein at least part of the inner surface of the core region is coated with a reactant for causing detectable optical changes in the analyte.

5 11. A sensor according to any one of the preceding claims further comprising a light detector arranged to receive light coupled from the waveguide.

12. A sensor according to any one of the preceding claims, further comprising an excitation source for exciting the analyte.

10

13. A sensor according to claim 12, wherein the excitation source comprises a light source arranged to couple light into the waveguide.

14. A sensor according to any one of the preceding claims, further comprising a coupler,  
15 arranged to couple light into and/or from the waveguide.

15. A sensor according to claim 14, wherein the coupler is arranged to couple light from a light source into a proximal end of the waveguide and also couple light from the proximal end of the waveguide into a light detector.

20

16. A sensor according to claim 15, wherein the coupler is a fused fibre coupler having first port coupled to the light source, a second port coupled to the light detector and a third port coupled to the proximal end of the waveguide.

25 17. A sensor according to any one of the preceding claims, further comprising a reflector that is arranged to reflect light that is propagating towards a distal end of the waveguide back towards a proximal end of the waveguide.

18. A sensor according to claim 17, wherein the reflector is arranged to selectively reflect  
30 light other than light from the light source.

19. A sensor according to any one of the preceding claims comprising plural core regions, at least one core region being a hollow core region.

35 20. A sensor according to claim 19, comprising plural hollow core regions.

21. A sensor according to claim 20, wherein at least part of the inner surfaces of plural hollow core regions are coated with a reactant for causing a detectable optical change in the analyte.

5 22. A sensor according to claim 21, wherein each of the core regions are coated with a different reactant for causing different detectable optical changes in the analyte.

23. A sensor according to any one of the preceding claims, wherein the matrix material comprises fused silica glass.

10

24. A sensor according to any one of the preceding claims, wherein the matrix material comprises only fused silica glass.

15 25. A sensor according to any one of the preceding claims, wherein the waveguide comprises an optical fibre.

26. A sensor according to any one of the preceding claims, wherein the analyte is a fluid.

20 27. A photonic crystal waveguide for use in an analyte sensing arrangement with an analyte having an analyte refractive index, the waveguide comprising a core region and a cladding region, the cladding region comprising a matrix material having a first refractive index and embedded along its length, parallel to a core region, an array of cladding holes surrounding the core region, the waveguide providing, at a sensing and/or excitation wavelength of light, an effective refractive index in the cladding region that is lower than the refractive index of  
25 the analyte such that the waveguide is index guiding with the analyte in the core region.

28. A photonic crystal waveguide as claimed in claim 27, wherein the core region is a least partially filled with analyte.

30 29. A method of sensing a property of an analyte including the steps:

introducing the analyte into at least a part of the core region of a photonic crystal waveguide as claimed in claim 27 or claim 28;

coupling light into or otherwise exciting the analyte; and

detecting light coupled from the waveguide.



1/9

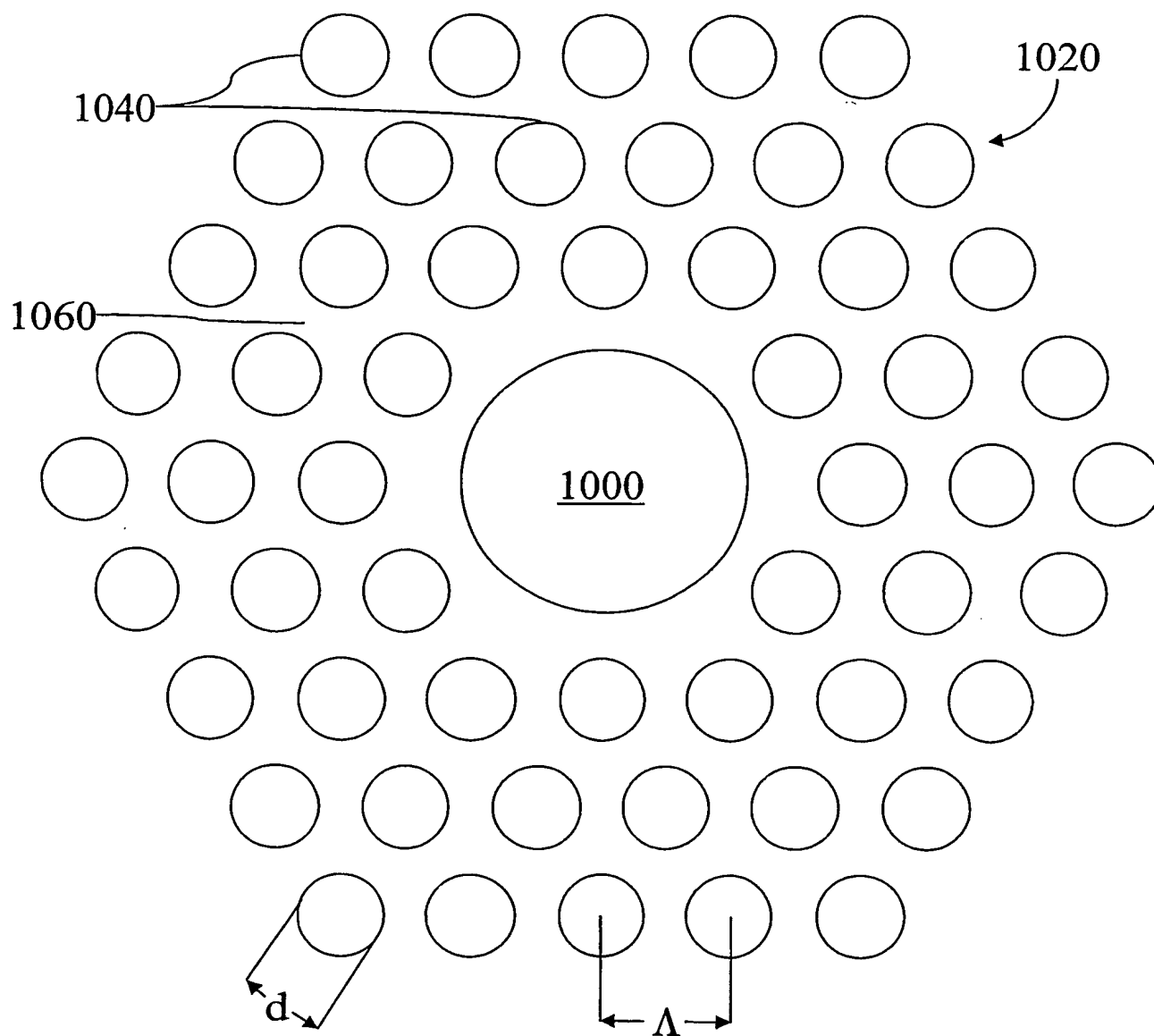


Figure 1

2/9

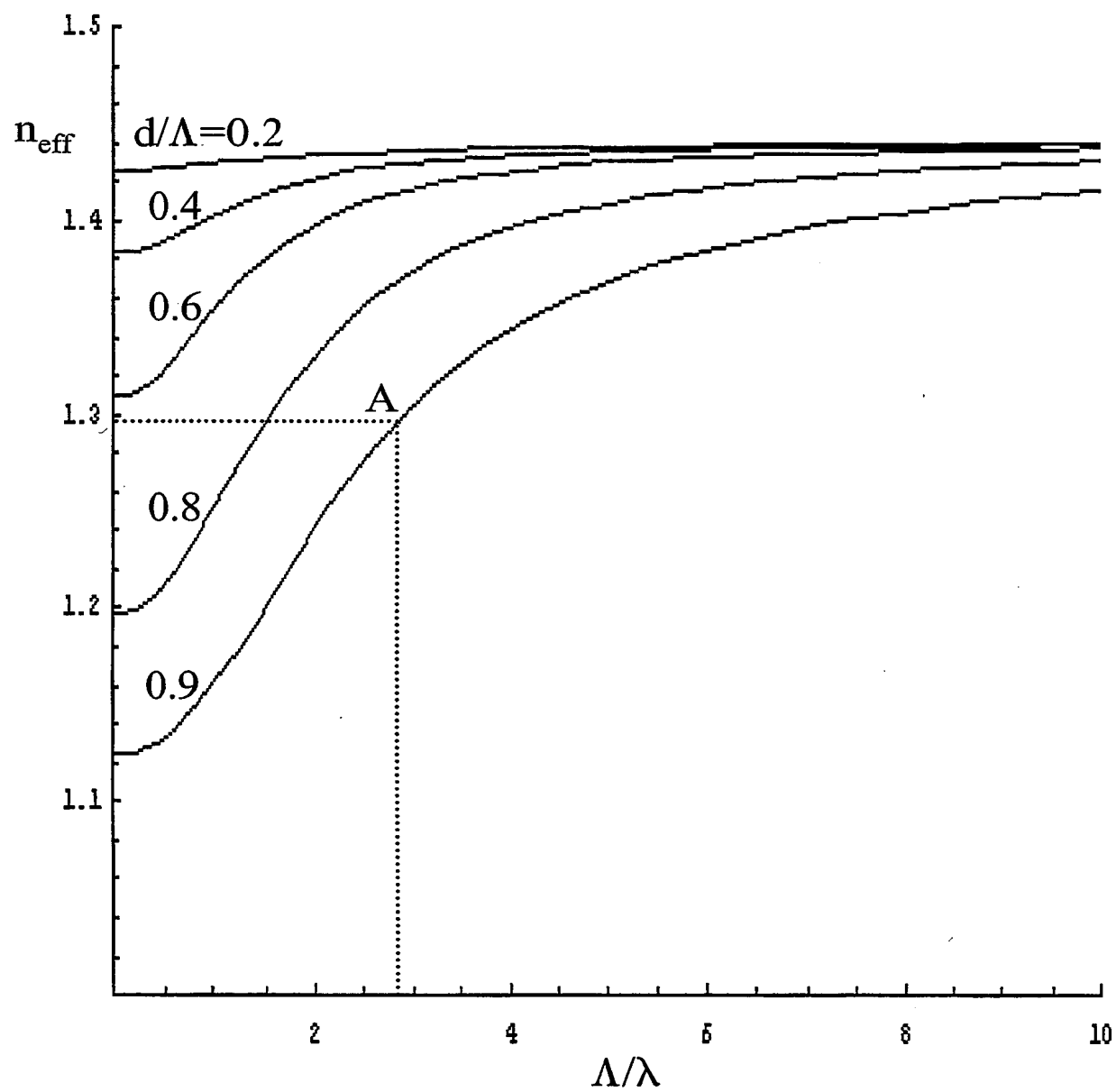


Figure 2

3/9

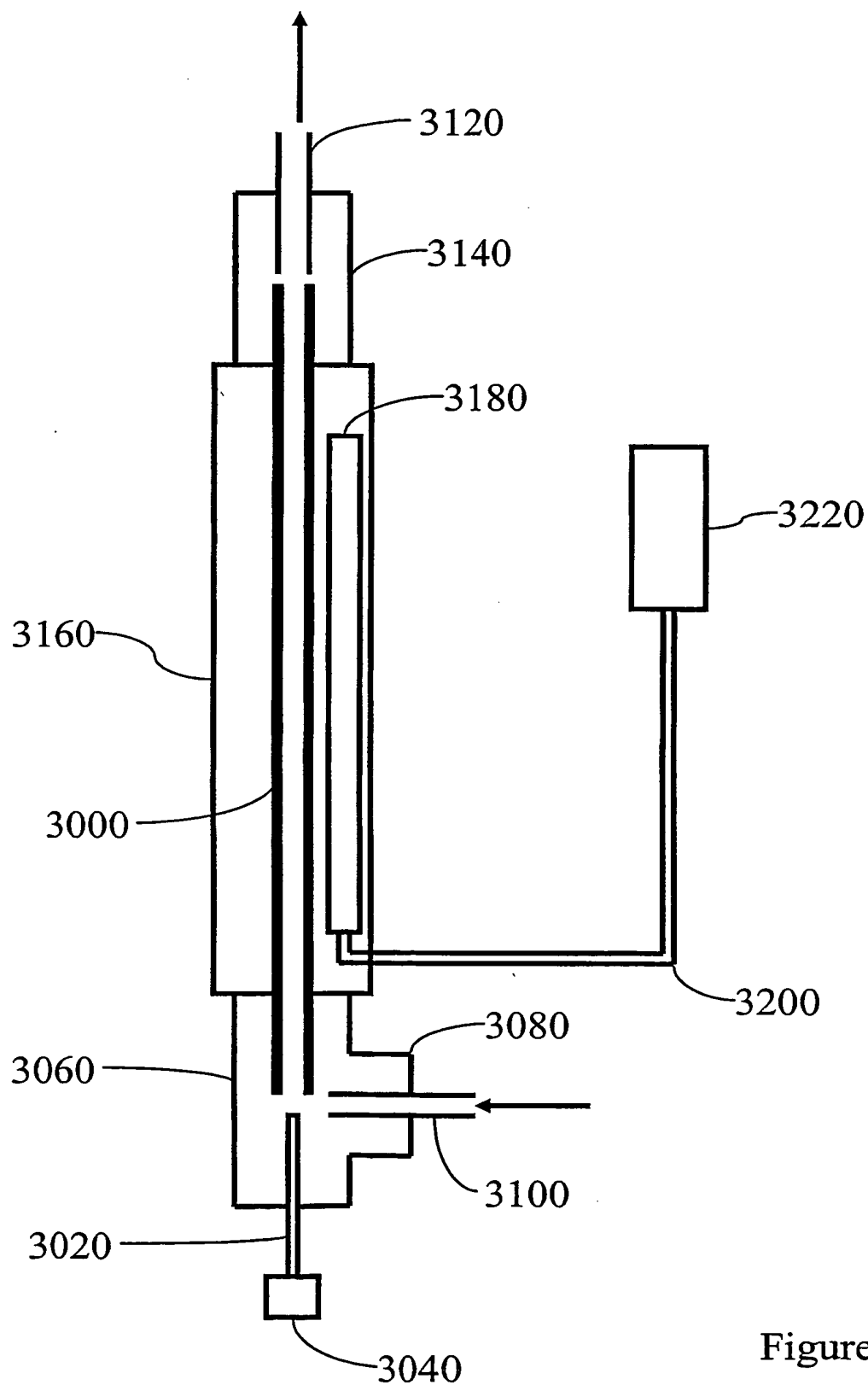


Figure 3

4/9

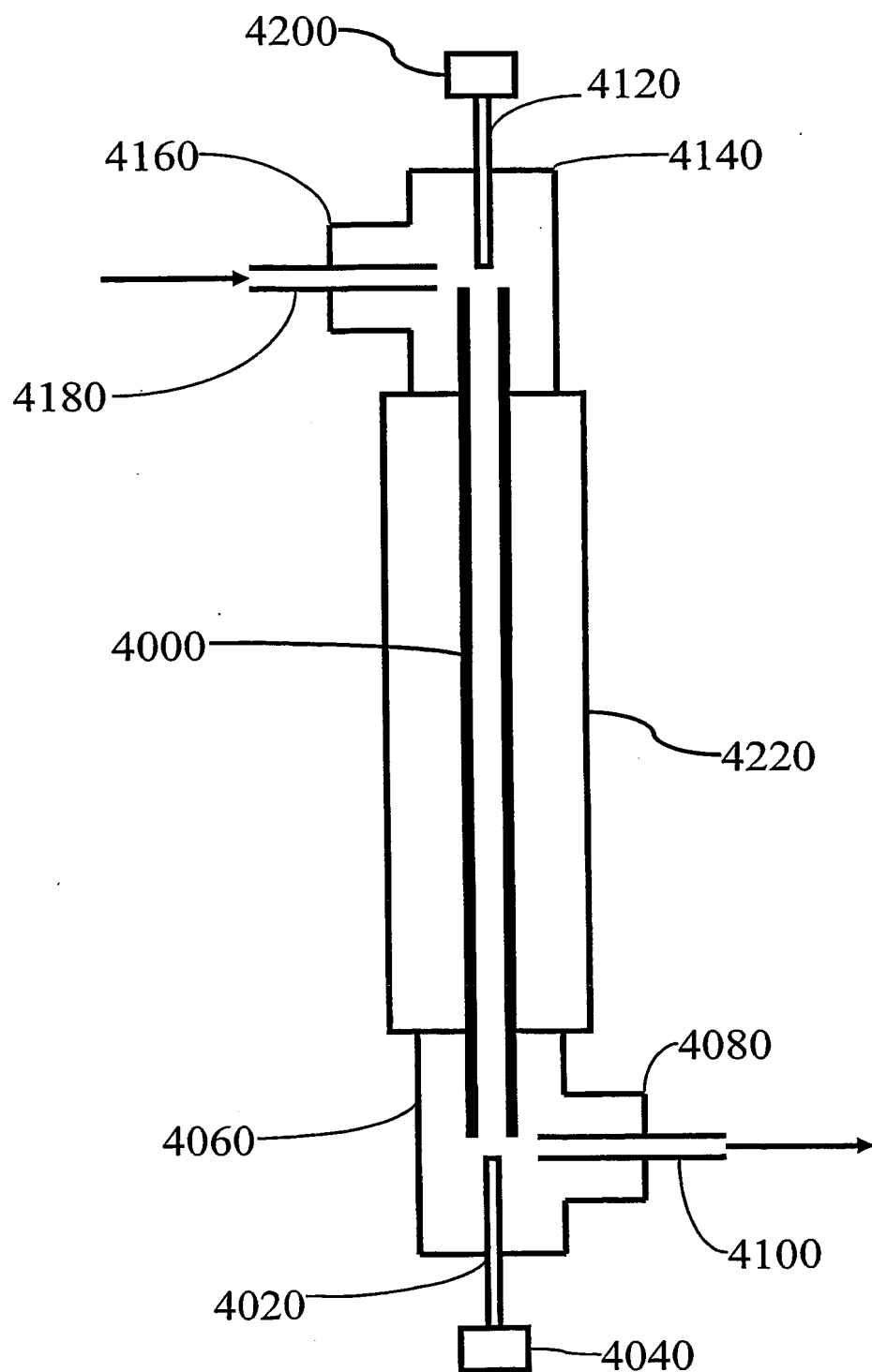


Figure 4

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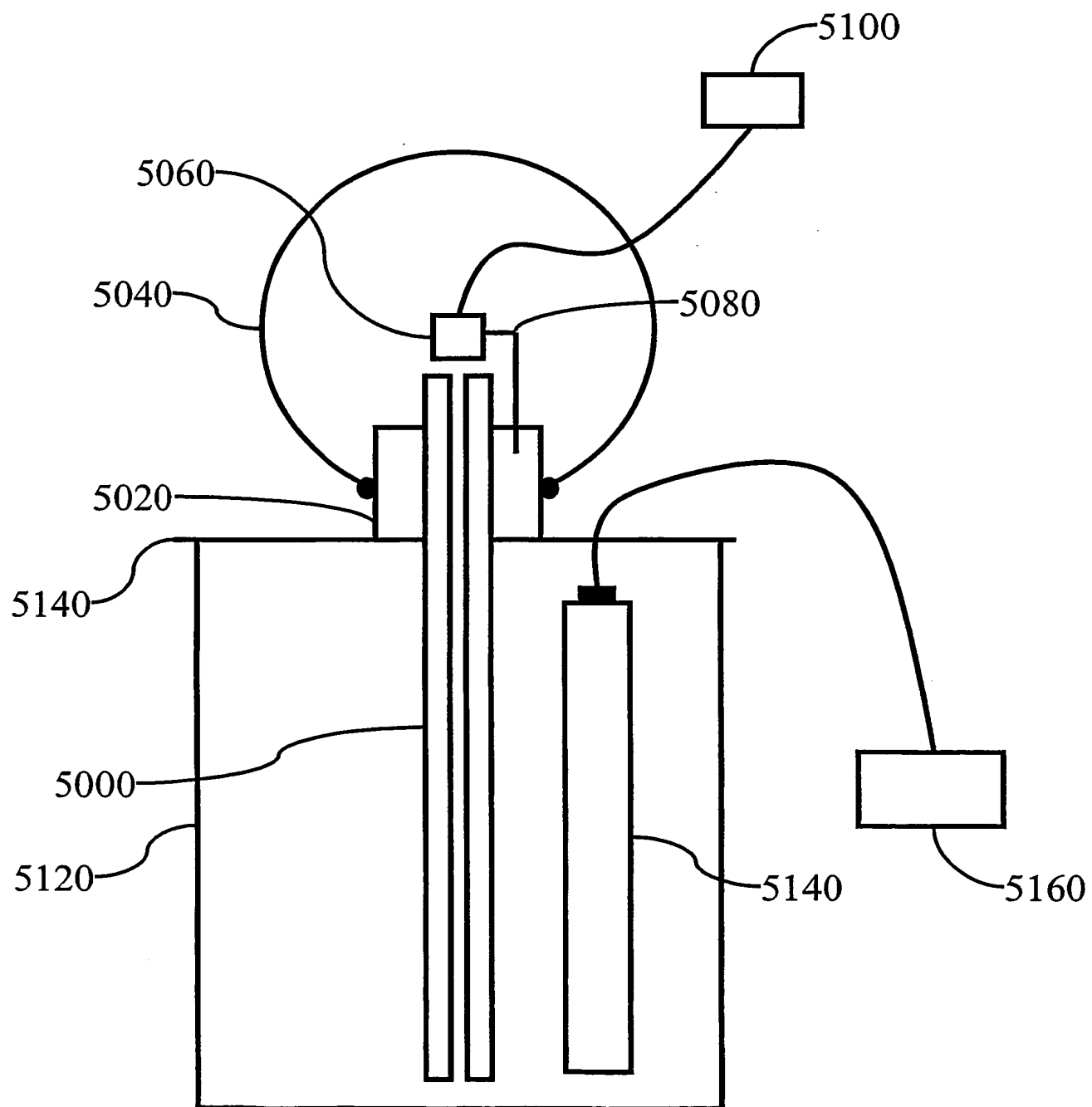


Figure 5

6/9

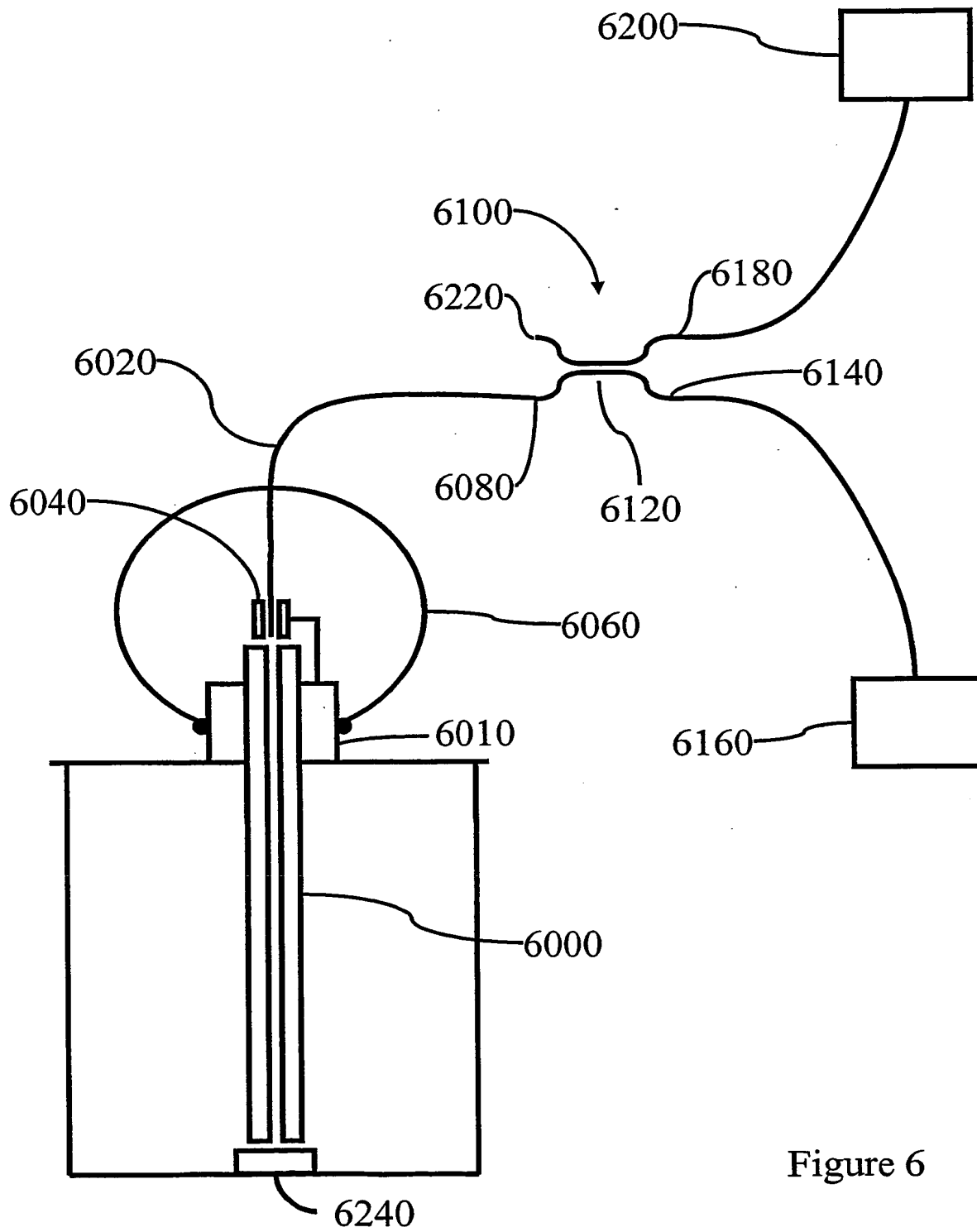


Figure 6

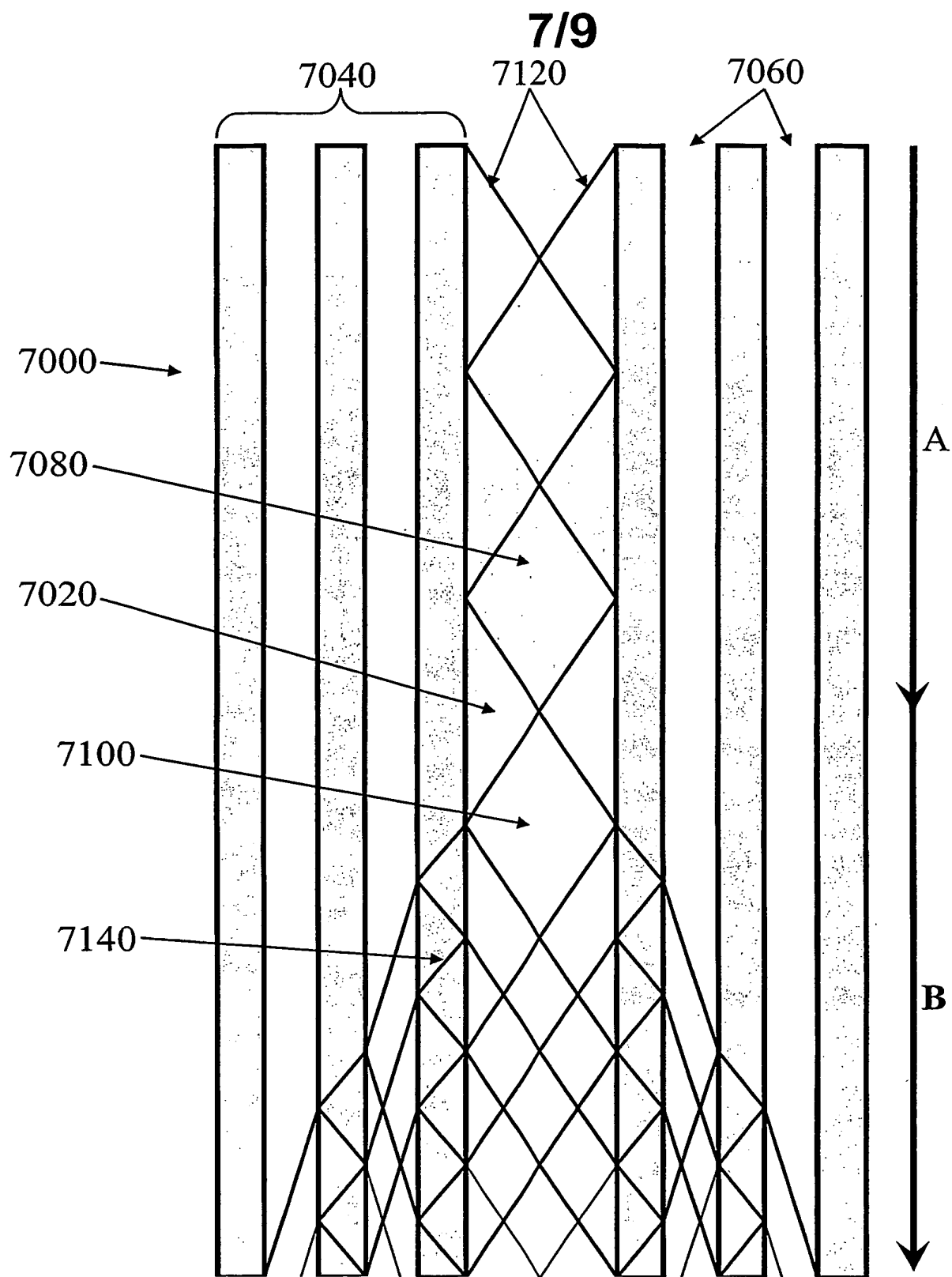


Figure 7

8/9

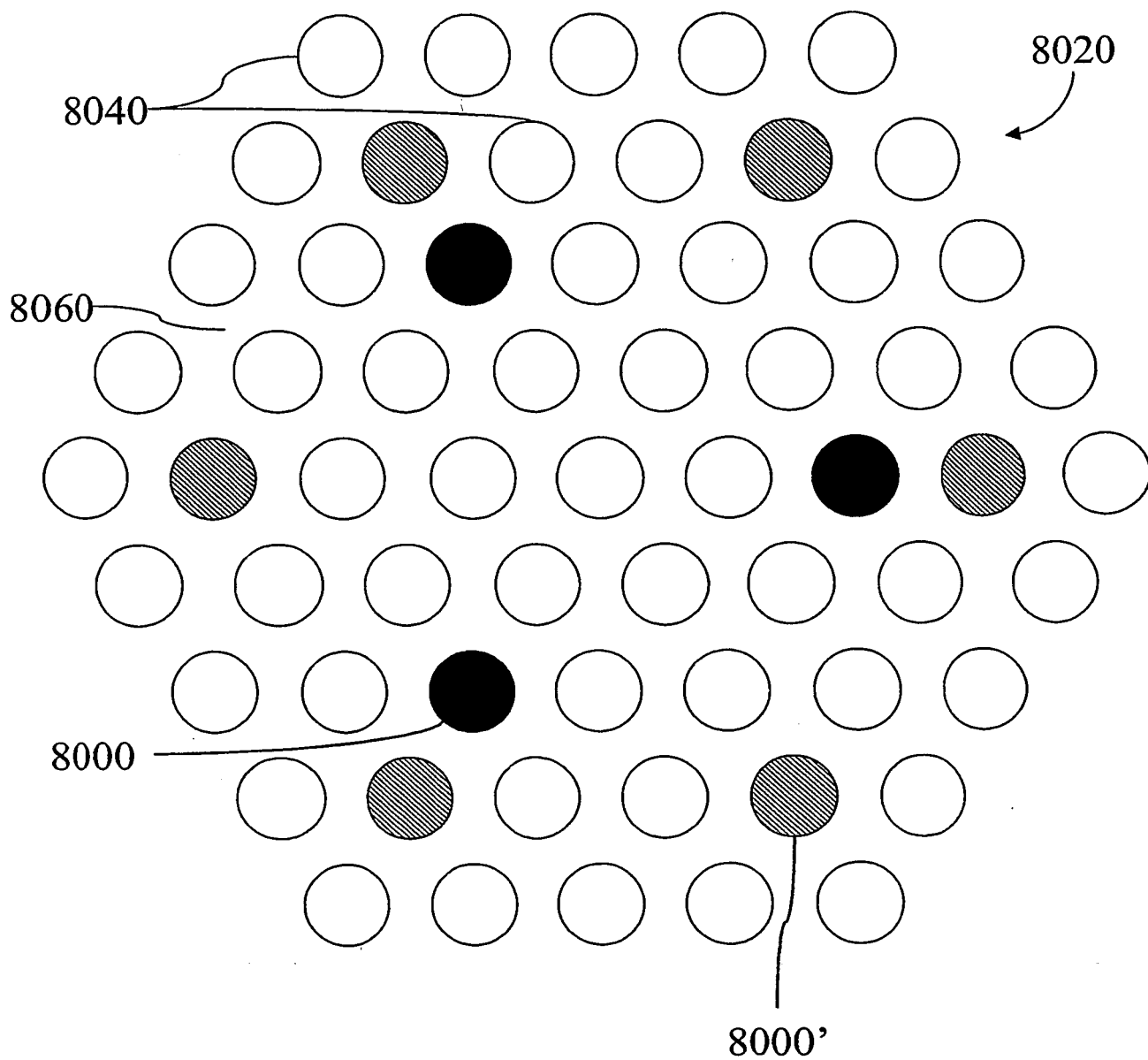


Figure 8



9/9

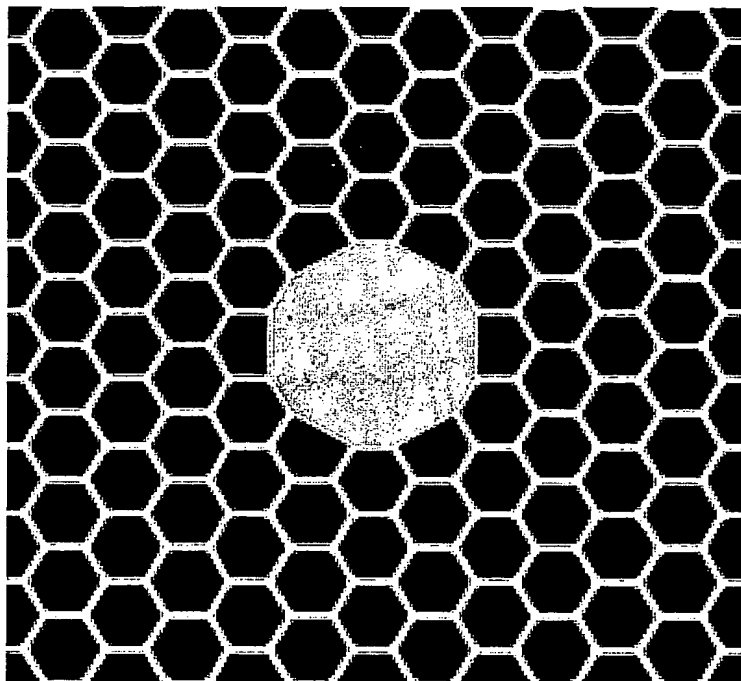


Figure 9

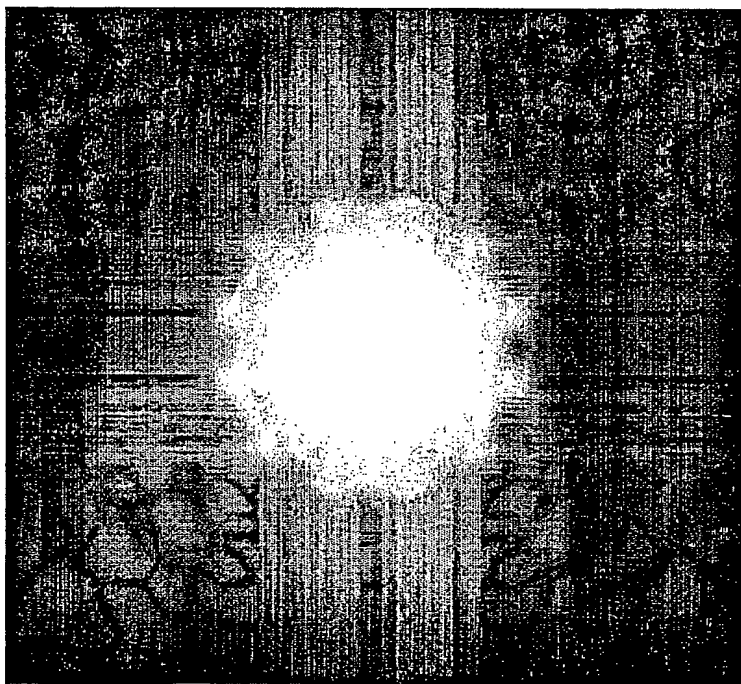


Figure 10

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 03/02727

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 G02B6/16 G02B6/20 G01N21/77

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 G02B G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, EPO-Internal, INSPEC

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 99 64903 A (BARKOU STIG EIGIL ;BJARKLEV ANDERS OVERGAARD (DK); BROENG JES (DK)) 16 December 1999 (1999-12-16) abstract	1-8, 10-14, 23-29
Y	page 18, line 5 - line 21 page 19, line 21 -page 20, line 30 --- -/--	9, 15-22

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

23 September 2003

Date of mailing of the international search report

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# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/GB 03/02727

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JONES J D C ET AL: "Photonic crystal fibres for sensor applications" 2002 15TH OPTICAL FIBER SENSORS CONFERENCE TECHNICAL DIGEST. OFS 2002(CAT. NO.02EX533), PROCEEDINGS OF INTERNATIONAL CONFERENCE ON OPTICAL FIBER SENSORS, PORTLAND, OR, USA, 6-10 MAY 2002, pages 565-568 vol.1, XP002255425 2002, Piscataway, NJ, USA, IEEE, USA ISBN: 0-7803-7289-1 the whole document	1-3, 27-29
Y	WO 02 42814 A (MAXWELL IAN ;UNIV SYDNEY (AU); FLEMING SIMON C (AU); EIJKELNBORG) 30 May 2002 (2002-05-30) abstract; figure 7 page 3, line 28 - line 34 page 4, line 11 - line 20	9
Y	US 2002/009719 A1 (HEALEY BRIAN G ET AL) 24 January 2002 (2002-01-24) abstract; figures 1-7,15	15-22
A	GB 2 350 904 A (SECR DEFENCE) 13 December 2000 (2000-12-13) abstract; figures 1,3	1-29

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 03/02727

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 9964903	A	16-12-1999	AU 755223 B2	05-12-2002
			AU 3026099 A	30-12-1999
			AU 755547 B2	12-12-2002
			AU 3810699 A	30-12-1999
			CA 2334510 A1	16-12-1999
			CA 2334554 A1	16-12-1999
			WO 9964904 A1	16-12-1999
			WO 9964903 A1	16-12-1999
			EP 1086393 A1	28-03-2001
			EP 1086391 A1	28-03-2001
			JP 2002517793 T	18-06-2002
			JP 2002517794 T	18-06-2002
			NZ 509201 A	26-11-2002
			US 6539155 B1	25-03-2003
WO 0242814	A	30-05-2002	WO 0242814 A1	30-05-2002
			AU 2328102 A	03-06-2002
US 2002009719	A1	24-01-2002	US 6406845 B1	18-06-2002
			AU 7287298 A	27-11-1998
			WO 9850782 A2	12-11-1998
GB 2350904	A	13-12-2000	AU 763796 B2	31-07-2003
			AU 3827400 A	23-10-2000
			CA 2368778 A1	12-10-2000
			CN 1353824 T	12-06-2002
			EP 1166160 A1	02-01-2002
			WO 0060388 A1	12-10-2000
			JP 2002541507 T	03-12-2002
			NO 20014740 A	03-12-2001
			PL 350990 A1	24-02-2003